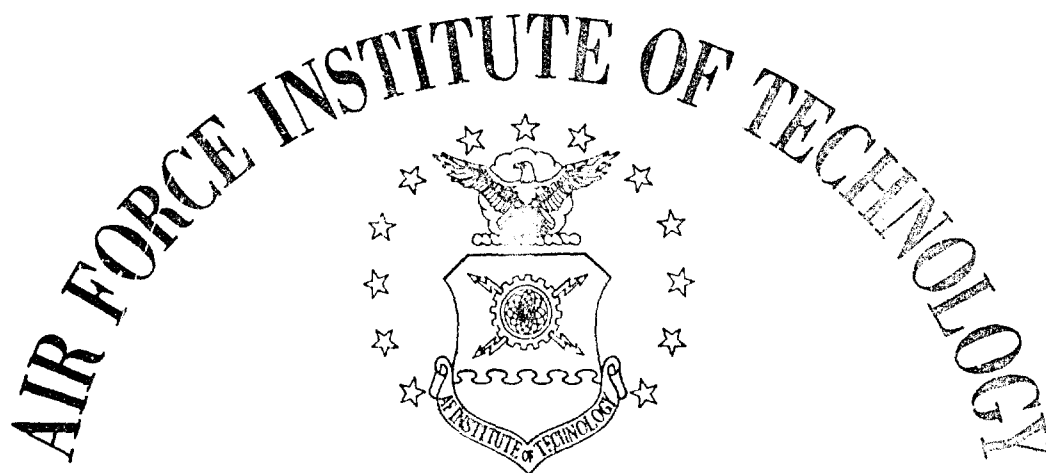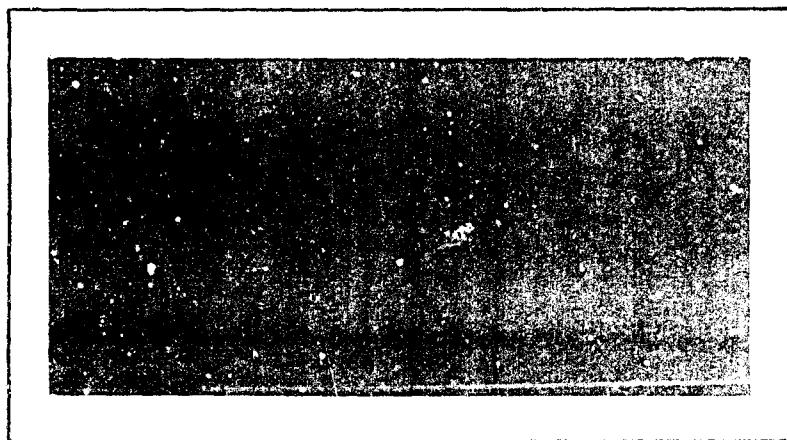AD NUMBER

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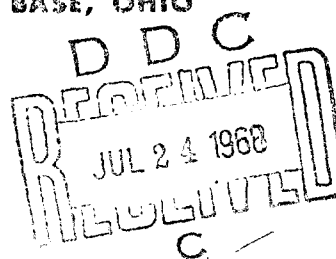
# AIR FORCE INSTITUTE OF TECHNOLOGY

## AIR UNIVERSITY
## UNITED STATES AIR FORCE

# SCHOOL OF ENGINEERING

WRIGHT-PATTERSON AIR FORCE BASE, OHIO

THE LATTICE PARAMETERS OF

SIX BINARY TITANIUM ALLOYS

AT CRYOGENIC TEMPERATURES

THESIS

GAW/MC/68-8   Roy G. Inness, Jr.
              Capt          USAF

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TITANIUM ALLOYS AT CRYOGENIC TEMPERATURES

THESIS

Presented to the Faculty of the School of Engineering of

the Air Force Institute of Technology

Air University

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Requirements for the Degree of

Master of Science

by

Roy G. Inness, Jr., B.S.

Capt                          USAF

Graduate Aerospace Weapons

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## Preface

This thesis presents the results of an experimental determination of the lattice parameters for six titanium alloys over the temperature range 300° to 5°K. It is hoped that this additional information, however small, will be of some value in the extensive investigation of the materials available for man's use.

It would have been impossible to complete this investigation without the aid and effort of many individuals. In acknowledgment, I wish to express my appreciation to the following people: Lt. Colonel Edward J. Myers, thesis advisor, for teaching me the fundamentals of x-ray diffraction and for his counsel and advise during this study; Dr. Harold Geoel, thesis sponsor, for making available the materials and equipment used in this study; Capt. Wilbur Simmons for his introduction to the computer program used; Mr. Paul Vincent for annealing the powder samples; Mr. Ken Storey for his timely supply of liquid helium; and Miss Sally Allen for typing this thesis.

Finally, my special thanks to Mr. Gene Charles whose operational knowledge of x-ray diffraction procedures and assistance in the day-to-day experiments were invaluable.

Roy G. Inness, Jr.

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## List of Symbols

$\overset{\bullet}{A}$ - Angstrom unit ($1 \times 10^{8}$ cm)

"a" - Lattice parameter (spacing between atoms in the basal plane)

"c" - Lattice parameter (spacing between basal planes)

$2\theta$ - Angle between the incident x-ray beam and the reflected x-ray beam

(hk·1) - Indices of a crystal plane (used to identify the recorded x-ray peaks)

Front-reflection region - $2\theta < 90°$

Back-reflection region - $2\theta > 90°$

## Abstract

The lattice parameters of six binary titanium alloys
(3.10Al, 4.99Al, 2.11V, 4.04V, 1.37Sn, and 2.56Sn) were
determined over the temperature range 300° to 5°K. $2\theta$
values were measured using the powder x-ray diffraction
technique; cryogenic temperatures were obtained by using
liquid helium and liquid nitrogen. Lattice parameters
were calculated by the analytical least-squares extra-
polation technique, using a computer program for the
actual calculations.

Both the "a" and "c" lattice parameters decreased
linearly from 300°K to a "break point" between 125° and
100°K. The "a" lattice parameter then continued to
decrease, at a reduced rate, from the "break point" to
5°K. Below the "break point" the "c" lattice parameter
either remained constant or continued to decrease at a
reduced rate, depending on the alloy.

The linear coefficients of thermal expansion were
calculated over the temperature ranges of 300° to 125°K
and 100° to 5°K.

THE LATTICE PARAMETERS OF SIX BINARY

TITANIUM ALLOYS AT CRYOGENIC TEMPERATURES

## I. Introduction

Frequently, requirements have often demanded that new materials be used operationally before a complete knowledge and explanation of their properties and behavior are known. Titanium alloys are now one of the most widely used of these new materials. This has led to an extensive research program to record and explain the properties and behavior of titanium, particularly at the extreme temperatures that it must withstand. A recent study has shown that there is a definite break in the approximately linear decrease of the lattice parameters of pure titanium, occuring near 100°K (Ref 3).

As part of the continuing investigation of titanium, at cryogenic temperatures, the primary purpose of this study was to determine the lattice parameters of six titanium alloys over a temperature range of 300° (room temperature) to 5°K. A secondary purpose was to determine if there was any correlation between the alloying element (amount and type) and the behavior of the lattice parameters at low temperatures. This study was conducted at the Air Force Materials Laboratory, Wright-Patterson AFB, Ohio.

Throughout this thesis, the x-ray peaks (lines) are identified by the (hk·l) value of the reflecting crystal plane, and all 2θ values are for resolved copper $K\alpha_1$ radiation. All lattice parameters are for the hexagonal crystal structure phase of titanium. Though some beta-titanium may have been present in the Ti-4.04V alloy, no x-ray peaks for this phase were observed.

This thesis is organized into five major sections: (1) Experimental Materials, (2) Experimental Equipment and Procedures, (3) Data Analysis, (4) Results, and (5) Conclusions. Supplementary information is included in the appendixes, and a list of symbols is included on page vi.

## II.  Experimental Materials

### Titanium Alloys

Six binary titanium alloy samples were supplied by
the Douglas Missile and Space Division of the Douglas Air-
craft Company.  The six alloys were, by weight percent:
Ti-3.10Al, Ti-4.99Al, Ti-2.11V, Ti-4.04V, Ti-1.37Sn, and
Ti-2.56Sn.  The iron, nitrogen, oxygen, and carbon con-
tents of each alloy were also furnished by Douglas and are
listed in Appendix C.  Each alloy was a high-purity
sample prepared from iodide titanium and was in sheet
form, approximately 0.035-inches thick.

### Preparation of Powder Samples

Powder samples, which expose many randomly-oriented
grains to the x-ray beam, will normally produce a satis-
factory x-ray pattern.  Therefore, an annealed 325-mesh
powder sample was prepared from each titanium alloy sheet.
The powder was prepared by first filing a sheet until a
sufficient quantity of filings was obtained.  A permanent
magnet was then passed over the filings to remove any par-
ticles of iron chipped from the file.  The filings were
then hand ground with a mortar and pestle until the result-
ing powder would pass through a 325-mesh screen.

The initial attempt to hand grind the filings indi-
cated that it would be a tedious and time consuming
process to grind a powder sample for each alloy.  Filings

from one alloy were then placed in a mechanical shaker
which utilized an iron cup and steel-ball bearings. After
a 15-minute run the magnet was passed over the resulting
powder, and it picked up almost all of the powder. It
appeared that most of the titanium had become imbedded on
the steel balls and the powder was mostly iron particles
from the cup, so this method of powder preparation was
discarded. Next, in an attempt to reduce the ductility of
the titanium, a strip was cut from one of the titanium
alloy sheets and cold-rolled until about a 50% reduction
in thickness was achieved. This reduced the ductility
enough to allow a sufficient quantity of powder from one
sheet to be prepared in about four hours, by hand grinding
filings from the cold-rolled strip in the mortar and
pestle.

A 325-mesh powder sample was then prepared from each
alloy sheet. The file was cleaned throughly with acetone
and a file brush after filing each alloy, and the mortar
and pestle were cleaned with hydrochloric acid to dissolve
any smeared titanium before grinding a different alloy.

A complete scan was then run on one of the unannealed
powder samples. All peaks above $2\theta = 90°$ were absent and
the peaks in the front-reflection region appeared weak
and broad. This resulted from the severe cold-working
during powder preparation. The Ti-1.37Sn powder sample
was then encapsulated in pure titanium foil and vacuum

annealed ($10^{-7}$ torr) for 10 hours at 650°C. A complete
scan of the annealed powder showed all peaks with an
intensity greater than five, as compared to an intensity
of 100 for the (01·1) peak, were present. However, the
peaks in the back-reflection region were still somewhat
broad, being up to one and one-half degrees wide. In an
attempt to increase their quality, the Ti-4.99Al powder
was annealed under the same conditions, but for 24 hours.
This longer annealing time resulted in such a minute
increase in quality that the remaining four powder samples
were vacuum annealed ($10^{-7}$ torr) for 10 hours at 650°C.
A higher annealing temperature was not tried because a
small amount of sintering occurred at 650°C. It was
known that sintering becomes a problem at higher tempera-
tures (Ref 3:6). Before annealing, each powder sample
was washed in acetone to remove any light foreign parti-
cles introduced during preparation.

Finally, after annealing, the diamond powder that was
needed to align the cryogenic attachment was added to each
powder sample. The diamond powder was obtained by washing
45-micron commercial diamond grinding paste in acetone
until the diamond powder separated. An approximate ratio
of one-part diamond powder to three-parts titanium powder
was used.

### III. Experimental Equipment and Procedures

#### Cryogenic Temperatures

A Materials Research Corporation Model X-86GC low-temperature, high-vacuum attachment, designed for use with the General Electric SPG spectrogoniometer, was used to obtain the cryogenic temperatures. It consists basically of an inner and outer dewar, a copper sample mounting stage, and a base unit (see Figure 8). A carbon-resistance thermometer and a copper-constantan thermocouple are built into the rear face of the copper stage for temperature measurements. It also has a built-in heating element for temperature variation. The base unit has provisions for translation along the x-axis and rotation about the y-axis and the z-axis. This allows the copper stage to be aligned with the focusing circle of the spectrogoniometer. The areas around the sample stage, between the two dewars, and around the outer dewar were evacuated to $10^{-5}$ torr.

A powder-sample temperature of 5°K was obtained by filling the outer dewar with liquid nitrogen and the inner dewar with liquid helium. 40°K was obtained by using the heater element. However, use of the heating element greatly increased the evaporation rate of the helium. This allowed only about 45 minutes at 5°K and 45 minutes at 40°K before all of the helium evaporated. This time was just sufficient to align the low-temperature attach-

ment and slow scan all the desired peaks. 85°K was obtained by filling both the inner and outer dewars with liquid nitrogen. The heating element was then used to obtain 100°, 125°, and 160°K. 200° and 250°K were obtained by removing the liquid nitrogen from the inner dewar and using the heating element.

The carbon-resistance thermometer was used to measure the 5°K temperature, and the copper-constantan thermocouple was used for 85°K and higher. Both devices were used for 40°K. The carbon resistance was measured with a Wheatstone bridge, and the thermocouple output by a Leads and Northrup 8686 millivolt potentiometer. The thermocouple reference junction that is located at the electrical connector pins on the base of the dewar is supposed to be at room temperature. However, cold liquid nitrogen gas that was boiling off from the outer dewar was cooling the reference junction. The actual temperature of the reference junction was determined by taping a laboratory-type mercury thermometer bulb next to the connector pins.

The 40°K temperature measurement was the most inaccurate, as the calibration curves for both temperature measuring devices are flat in this range (Ref 3:30). The two temperature-measuring devices were within 3°K of each other for this temperature, and it is considered accurate to ±2°K. All other temperatures are considered accurate

to $\pm 1°K$, being limited by the accuracy of the reference junction temperature. All temperature readings were recorded to the nearest degree.

## X-Ray Diffraction

A General Electric XRD-5 x-ray unit, SPG spectrogonio-meter, and SPG-3 detector were used. The basic spectro-goniometer is considered accurate to $\pm 0.002°$. The detector unit utilizes a xenon-filled proportional counter tube, and the diffraction peaks were recorded on a G.E. millivolt recorder strip chart. Nickel filtered copper radiation, with a $K\alpha_1$ wavelength (lamda) of $1.54050\text{Å}$, was used. The x-ray tube was operated at 50,000 volts and 31 milliamps for all runs.

A powder sample was mounted on the copper sample stage face by using an acetone slurry and rotating the stage until the sample spread evenly. After the desired temperature was obtained, the cryogenic attachment was aligned. All alignment was done using the diamond (111) $K\alpha_1$ peak located at $2\theta = 43.93°$. This $2\theta$ value was cal-culated using a diamond lattice parameter value of $3.5667\text{Å}$ (Ref 1). Since the $2\theta$ shift for this diamond peak is only about $+0.006°$ at $5°K$ (Ref 5), $43.93°$ was used for align-ment at all temperatures. Alignment was accomplished by setting the diffractometer to $43.93°$ and adjusting the axes controls of the X-86GC base until the recorder pen peaked out. The diamond peak was then slow scanned using

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a high-resolution technique (1° beam slit and 0.05° detector slit) and a 0.5-second time constant. The alignment was accepted if the diamond peak was located within ±0.01° of 43.93°. After alignment the axes-locking screws on the X-86GC base were tightened. This prevented any minor rotation of the cryogenic attachment about the z-axis because of the tension of the vacuum line as the diffractometer was scanning. Each time the temperature was changed the cryogenic attachment was re-aligned.

Because of the time limitation at low temperatures and the need to scan as many peaks as possible for the extrapolation technique, a schedule of time constants, beam and detector slits, scanning speeds, and peaks of acceptable quality was determined after considerable experimentation at room temperature. The eight best peaks above $2\theta = 60°$ were determined and scanned for all samples, except for the (21·3) peak of the titanium-aluminum alloys, which merged with the (331) diamond peak. As the $2\theta$ values of the peaks increased, the time constant had to be increased in order to get a smooth trace with the recorder pen. A 2-second time constant was used for the (11·0), (10·3), (11·2), and (20·1) peaks, a 4-second time constant for the (20·3) and (21·1) peaks, and a 8-second time constant for the (11·4) and (21·3) peaks. In the back-reflection region a low-resolution

technique (3° beam slit and 0.2° detector slit) was re-
quired to obtain satisfactory intensity of the x-ray
peaks. The increased quality of the peaks in the front-
reflection region allowed use of a higher-resolution
technique (3° beam slit and 0.1° detector slit). All
peaks were scanned using a 0.4° per minute diffractometer
scanning speed and 500 counts per second on the recorder
scale.

The selected peaks for each alloy were scanned, using
the above conditions, at nine selected temperatures. The
2θ values were then determined from the strip-chart
record and the appropriate time-constant correction (dis-
cussed on page 11). Appendix A lists the corrected 2θ
values for each alloy and temperature.

## IV.  Data Analysis

### Time-Constant Correction

All measured 2θ values were corrected for the time-
constant lag in the recorder.  The correction, for each
time constant used, was determined by slow scanning one
of the diamond peaks backward and forward seven times.
By comparing the mean 2θ value for the forward scans with
the mean 2θ value for the backward scans, the time-constant
lag for a forward scan only was determined.  The resulting
corrections for a 2, 4, and 8-second time constant were
0.02°, 0.03°, and 0.04°, respectively.  The time-constant
correction was added to the measured 2θ value.

### Calculation of Lattice Parameters

There are two basic methods for calculating accurate
lattice parameters.  Both have been developed to elim-
inate most of the systematic errors in the measured 2θ
values, such as absorption, eccentricity, and divergence.
The first method uses an internal standard to obtain the
2θ correction factors for systematic errors.  The second
method extrapolates the lattice parameter values to θ =
90°, where theoretically all systematic errors vanish.
In the second method, the values of the computed lattice
parameters are plotted as the ordinate, and the abscissa
is the corresponding value of a trigonometric function of
θ.

A simple and very direct internal-standard method is to use the diamond powder, that was added to each alloy powder sample, as an internal standard (Ref 3:12). Using this technique, only two titanium peaks and one diamond peak (located near each other in the back-reflection region) have to be scanned. By comparing where the diamond peak is located relative to where it should be located, the systematic errors in the titanium $2\theta$ values can be rapidly determined. This method also alloys "a" and "c" to be readily calculated from a simultaneous solution of the hexagonal interplanar spacing equations for the two titanium peaks scanned. However, this method was not used because of the following reasons: (1) the poor quality of the $(30 \cdot 2)$ titanium peak located near 149°, (2) the merging of the $(21 \cdot 3)$ peak of the titanium-aluminum alloys with the (331) diamond peak, rendering the $(21 \cdot 3)$ peak useless for measurements, (3) the doubt about the actual shift of the (331) diamond peak below 100°K (Ref 5).

Therefore, Cohen's extrapolation method was used to calculate the lattice parameters (Ref 4:467). This method is an analytical, least-squares extrapolation of "a" and "c" to $\theta = 90°$. A trigonometric function of $\theta$, rather than $\theta$ itself, is used to provide for linear extrapolation. The extrapolation to $\theta = 90°$ eliminates most of the systematic errors, while the least-squares method mini-

mizes the random errors. While Cohen's method will yield
excellent results, the number of calculations becomes
overwhelming when a large number of "a" and "c" values
are determined. This was overcome by using, a readily
available, but apparently seldom used, computer program
to determine the lattice parameters (Ref 6). This program
is a computerized version of Cohen's method. Inputs are
the measured $2\theta$ values, the corresponding (hk·l) values,
the desired weighting for each $2\theta$ value, the wavelength
of the radiation used, the type of crystal system, and
the desired extrapolation function. Outputs are the
values of the lattice parameters and the probable error
for each lattice parameter.

Once the raw data ($2\theta$ values) are measured, the only
variables to be selected by the user of the program are the
extrapolation function and the weight given to each $2\theta$
value. Two of the more commonly used extrapolation func-
tions are:

$$\cos^2\theta \qquad \text{(Bradley and Jay)} \quad (1)$$

$$\frac{1}{2}\left(\frac{\cos^2\theta}{\sin\theta} + \frac{\cos^2\theta}{\theta}\right) \quad \text{(Nelson and Riley)} \quad (2)$$

henceforth referred to as $F_1$ and $F_2$ respectively. When
peaks in the front-reflection region are used, $F_2$ will
usually result in a more accurate extrapolation of the
lattice parameters (Ref 4:464). However, since these
extrapolation functions were developed for the Debye-
Scherrer photographic method and the computer program

allowed rapid determination of the lattice parameters, a comparison of $F_1$ and $F_2$ was undertaken. The room temperature lattice parameters of Ti-4.99Al, Ti-1.37Sn, and Ti-2.56Sn, and the lattice parameters of Ti-3.10Al from 300° to 5°K were calculated using each of the above extrapolation functions.

All "a" and "c" values calculated with $F_2$ were consistently 0.0002Å to 0.0003Å lower than those calculated with $F_1$. However, the probable error using $F_1$ was always lower, usually about 0.0002Å, than the probable error using $F_2$. This lower probable error indicated that the extrapolation was more linear when $F_1$ was used. All lattice parameters in this thesis were calculated using $F_1$.

Initially, a weight of one was used for each $2\theta$ value. This led to slightly erratic results, since a small random error in the measured $2\theta$ value for a low-angle peak seriously affected the extrapolated lattice parameter. (Ref 2:324). This effect was partially eliminated by giving a weight of two to all $2\theta$ values in the back-reflection region and a weight of one to all $2\theta$ values in the front-reflection region. Thus, the lattice parameters were calculated using a computerized version of Cohen's analytical least-squares extrapolation method, with $\cos^2\theta$ as the extrapolation function and a double weight given to the back-reflection $2\theta$ values. All com-

puter runs were done on an IBM 7094 computer.

## Accuracy

The main items that must be considered in assessing the accuracy of the computed lattice parameters are: (1) the diffractiometer, (2) alignment of the cryogenic attachment, (3) temperature measurements, (4) quality of the peaks, (5) alloy analysis, and (6) the probable error output of the computer program. The first three items were considered in Section III. The quality of the peaks varied progressively, from excellent in the low angle front-reflection region to poor in the extreme back-reflection region. Appraising the combined effect of the first four items, the measured $2\theta$ values are considered accurate to $\pm 0.01°$ in the front-reflection region and $\pm 0.02°$ in the back-reflection region.

Since the chemical analysis of each alloy was done before the powder samples were prepared, the exact chemical analysis of the powder samples is really unknown. All practical care was taken to prevent any contamination (especially by oxygen and nitrogen) during the preparation of the powder samples. However, the observation that the calculated room temperature "c" values for all six titanium alloys were larger than most previously reported values (see Appendix E) indicates that there may have been a small amount of oxygen and/or nitrogen contamination in the powder samples. It is known that only 0.5 weight per

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cent oxygen increases the value of the "c" lattice param-
eter by approximately 0.008Å (Ref 7:1010).

For each lattice parameter calculated, the computer
program also calculated a statistical probable error.
The probable errors are listed in Appendix B. This
probable error was a 50% confidence interval, defined
(for a normalized distribution) as 0.675 times the stand-
ard deviation. The mean of the probable errors for all
"a" lattice parameter values calculated was ±0.0004Å.
The mean of the probable errors for all "c" lattice
parameter values calculated was ±0.0007Å.

## V. Results

### Discussion

The calculated lattice parameters for each alloy, plotted as function of temperature, are presented in Figures 1 to 6. The c/a ratios, calculated from values taken from the curves, are presented in Figure 7.

The calculated room temperature "a" value for each of the six titanium alloys was in good agreement with previously reported values. The calculated room temperature "c" value for each of the six titanium alloys was somewhat larger than most previously reported values, possibly due to oxygen and/or nitrogen contamination during preparation of the powder samples. Appendix E contains a comparison of the room temperature lattice parameters calculated in this thesis with previously reported values.

Appendix E also indicates the effect of alloying on the room temperature lattice parameters. By comparing the atomic radius of titanium with that of aluminum, tin, and vanadium it can be readily seen that the size factor is primarily determining whether the lattice parameters of pure titanium are increased or decreased when alloyed with these elements. However, the effect of relative valence and electronegativity must also be considered to explain any deviation from Vegard's Law (varying in linear proportion to size (Ref 7:23).

Essentially, the lattice parameters of all six alloys
behaved the same at low temperatures. Both "a" and "c"
decreased linearly to a "break point" between 125° and
100°K. Below the "break point" the "a" lattice param-
eter continued to decrease, at a reduced rate, for all
alloys. Below the "break point" the "c" lattice parameter
either remained constant or continued to decrease at a
reduced rate, depending on the alloy.

The "break point" between 125° and 100°K and the
somewhat unusual behavior of the "c" lattice parameter
below the "break point" confirms the results of Ebneter
(Ref 3). While the exact cause of the "break point"
and the somewhat unusual behavior of the "c" lattice
parameter below the "break point" is unknown, Zwikker
relates this type of behavior to the values and behavior
of the Debye characteristic temperature, the low tempera-
ture specific heat constant, and lattice vibrations
(Ref 8:144-167).

Fig. 1. Lattice Parameters for Ti-2.11V Alloy

Fig. 2. Lattice Parameters for Ti-4.04V Alloy

Fig. 3. Lattice Parameters
for
Ti-1.37Sn Alloy

Fig. 4. Lattice Parameters for Ti-2.56Sn Alloy

Fig. 5. Lattice Parameters for Ti-3.10Al Alloy
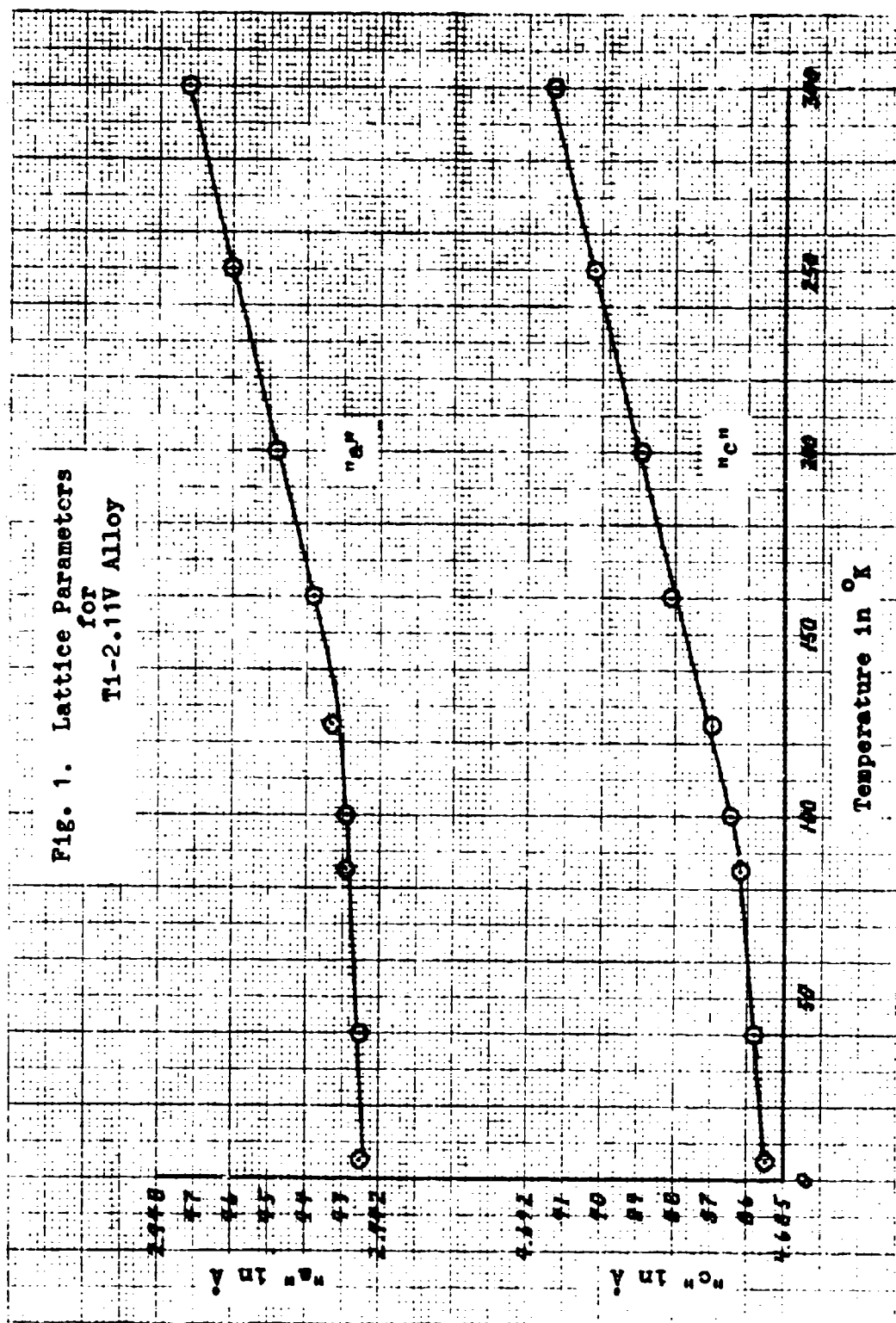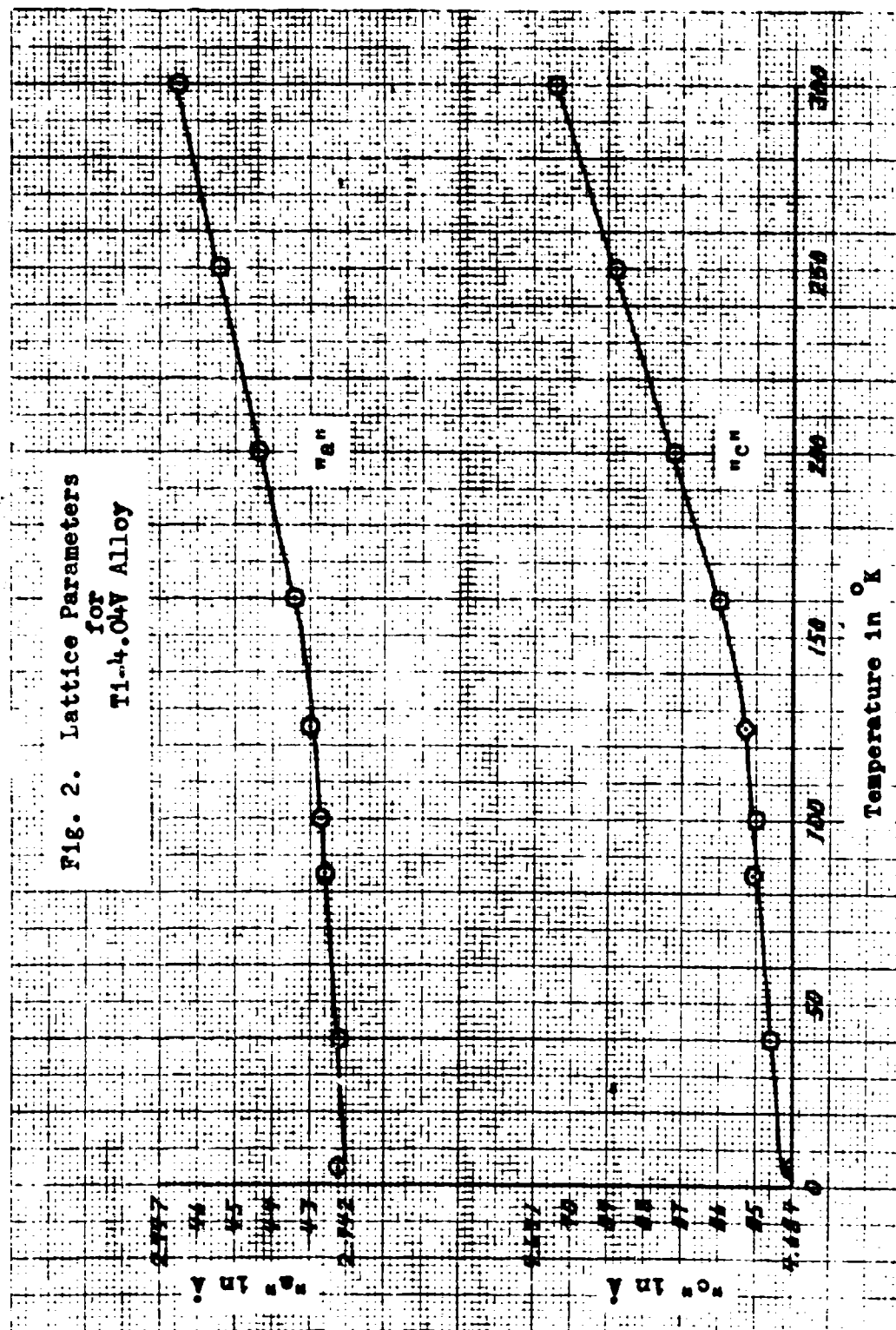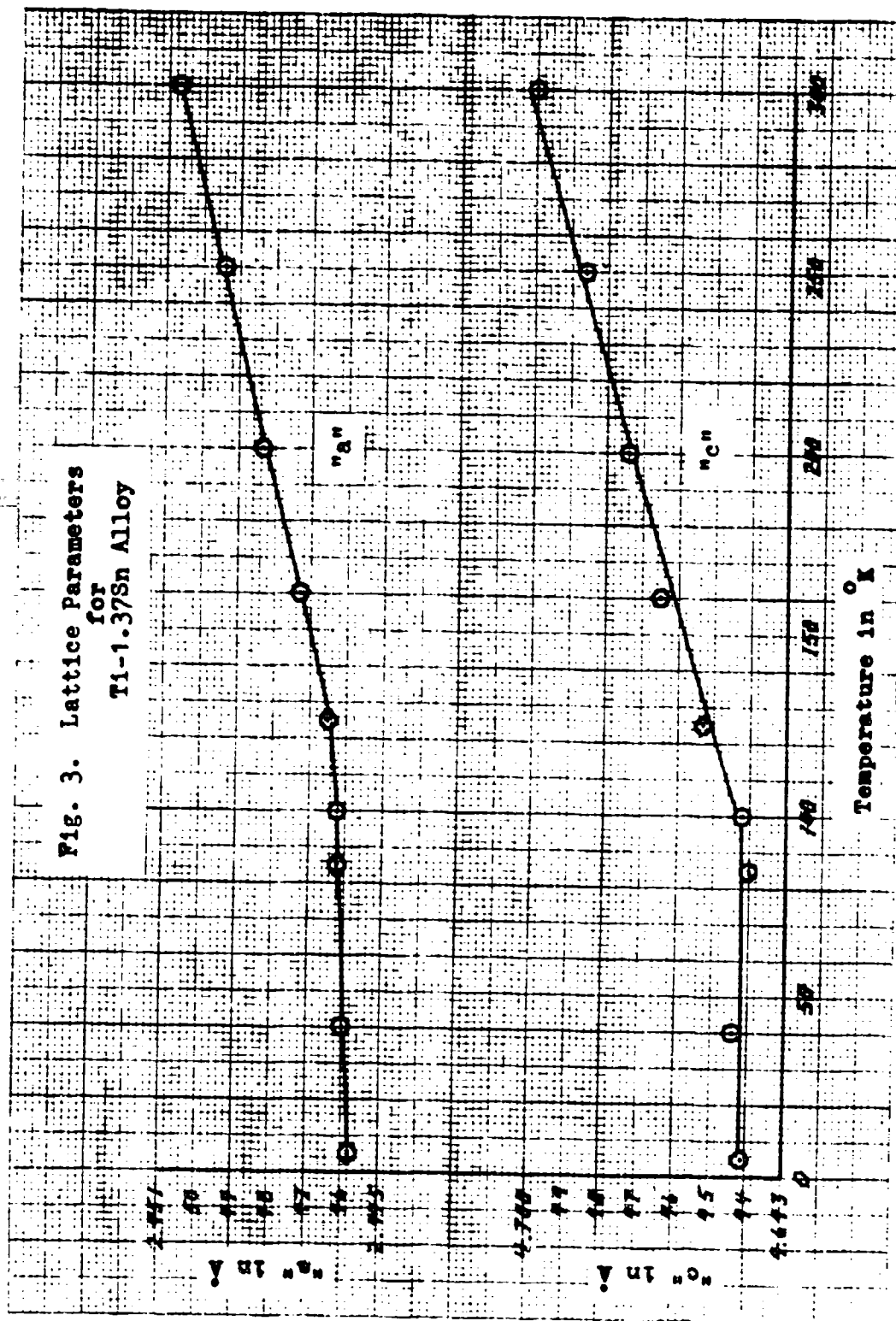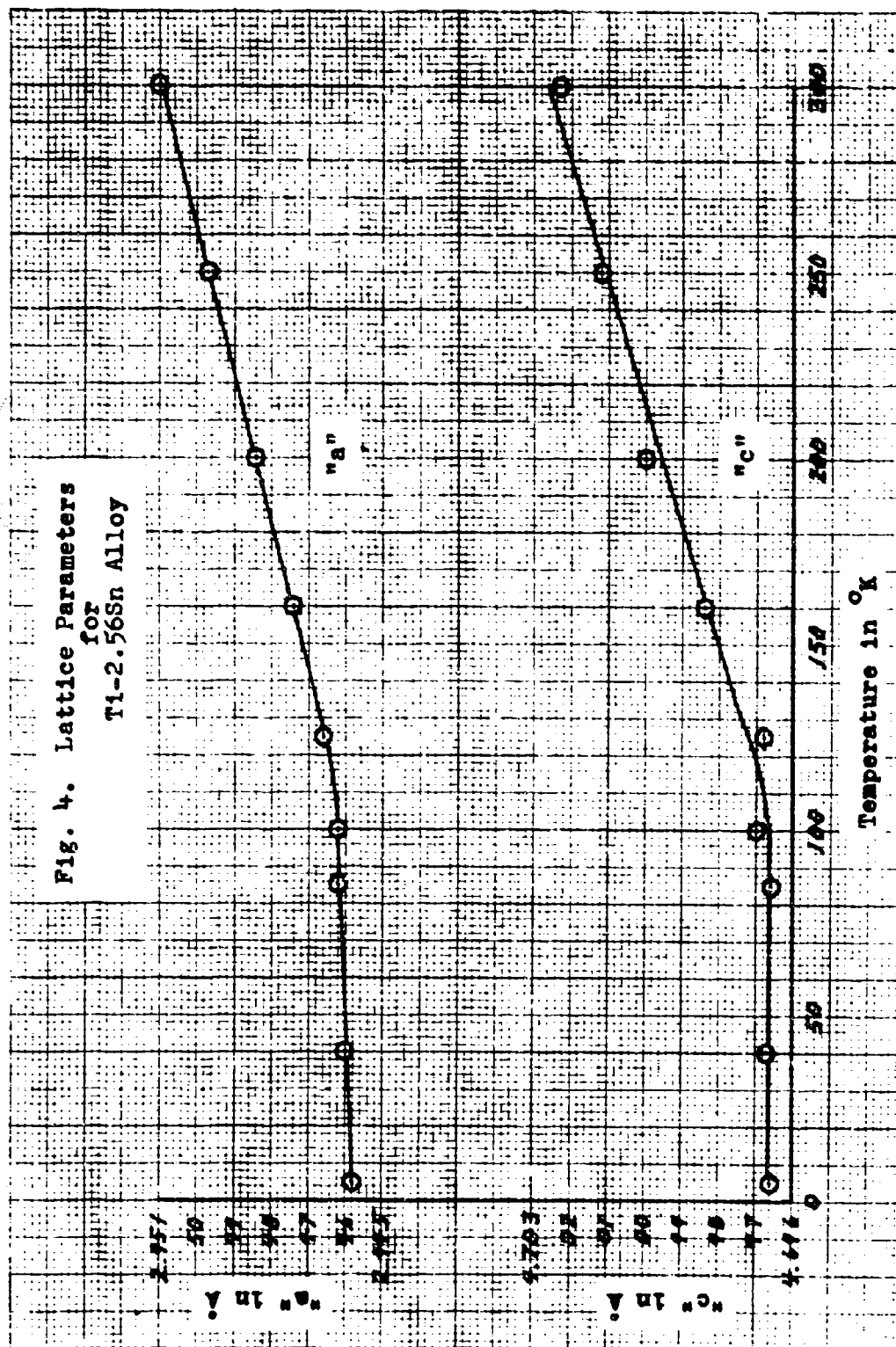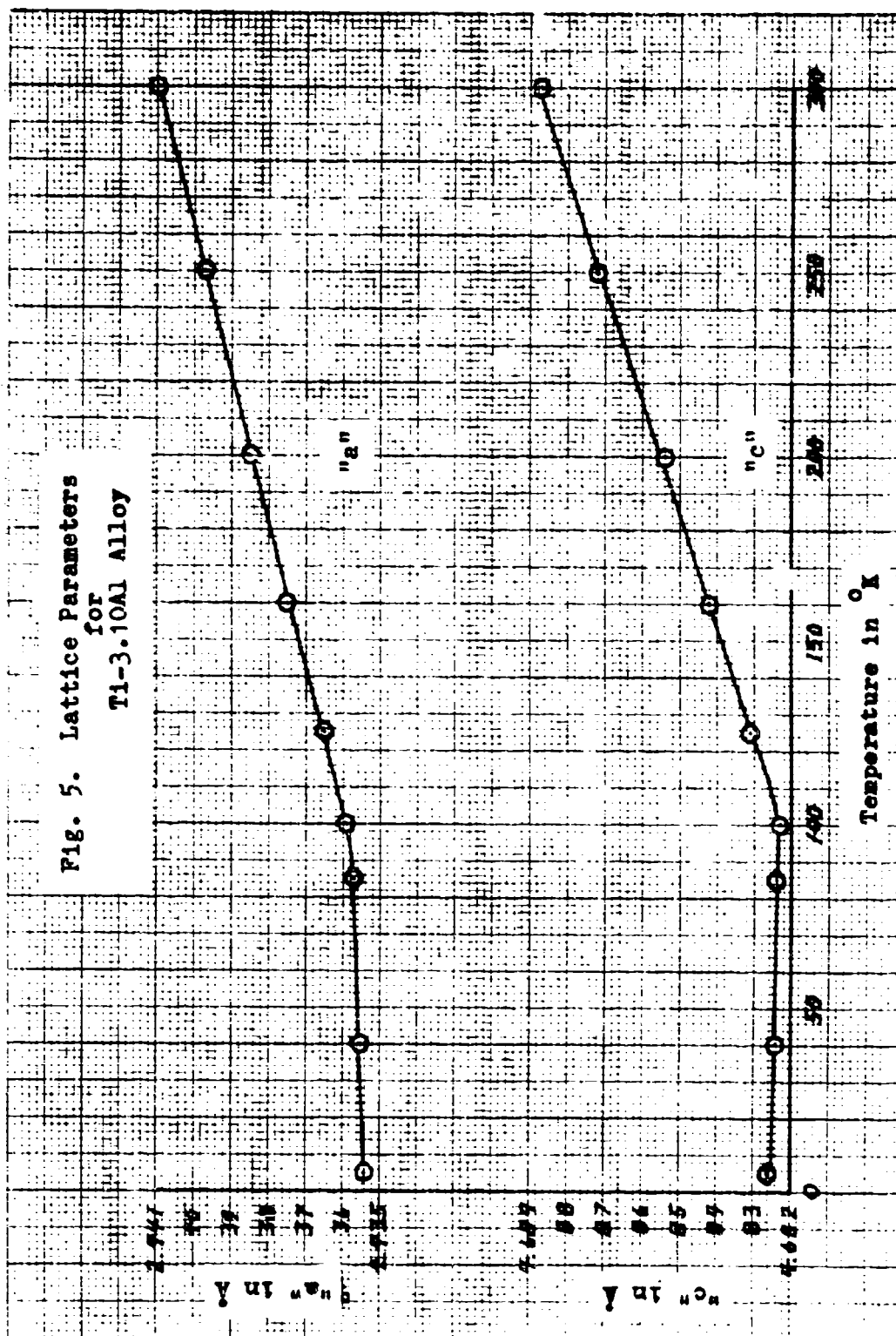
Fig. 6. Lattice Parameters for Ti-4.99Al Alloy

Fig. 7. c/a Ratios for Titanium Alloys Studied
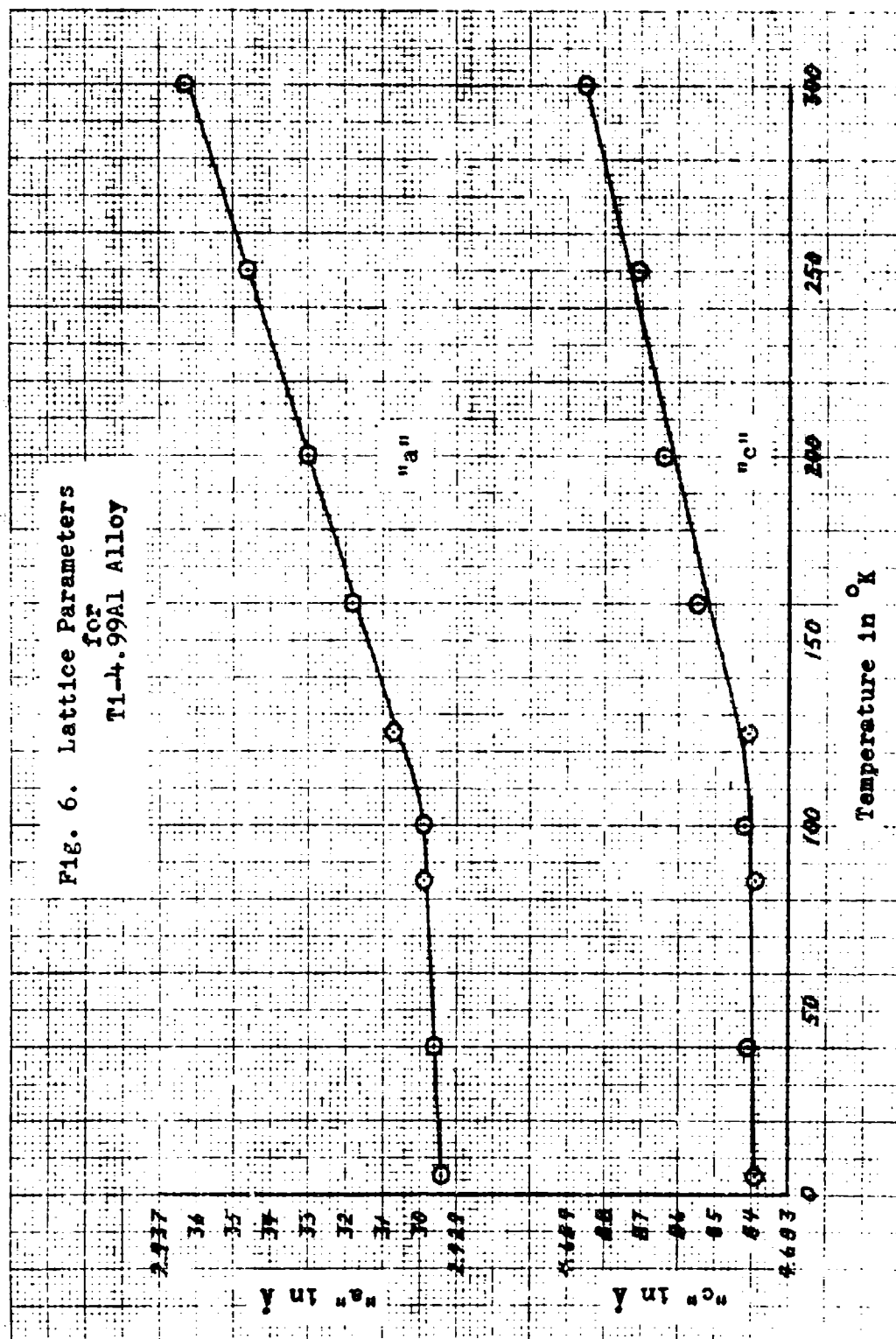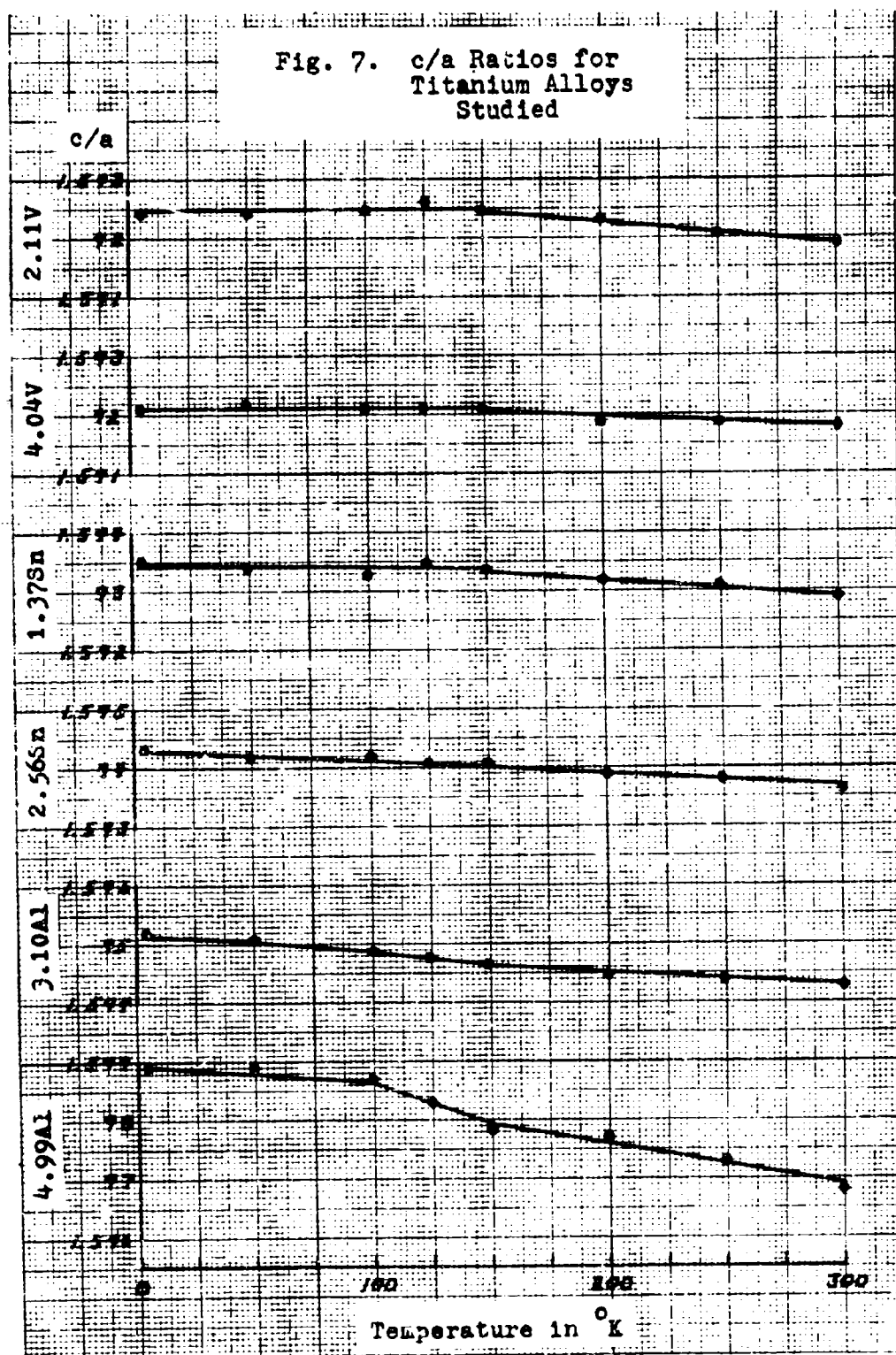
## Coefficients of Expansion

A linear coefficient of thermal expansion was calculated for each alloy over the temperature ranges of 300° to 125°K and 100° to 5°K using the expression:

$$\alpha = \frac{1}{T_1 - T_2} \left\{ \frac{2\Delta a}{3 \; a} + \frac{\Delta c}{3c} \right\} \qquad (3)$$

where $\alpha$ = linear coefficient of thermal expansion (per °C)

$T_1$ = upper temperature in °K

$T_2$ = lower temperature in °K

$a$ = lattice parameter "a" at temperature $T_1$

$\Delta a$ = change in lattice parameter "a" from $T_1$ to $T_2$

$c$ = lattice parameter "c" at temperature $T_1$

$\Delta c$ = change in lattice parameter "c" from $T_1$ to $T_2$

The results are presented in the table below.

### Table I

### Linear Coefficients of Thermal Expansion

### (Multiply by: $10^{-6}$/°C)

| Alloy | 300° to 125°K | 100° to 5°K |
|-------|---------------|-------------|
| 2.11V | 7.1 | 1.9 |
| 4.04V | 6.9 | 2.5 |
| 1.37Sn | 7.4 | 1.0 |
| 2.56Sn | 7.8 | 1.0 |
| 3.10Al | 8.1 | 1.1 |
| 4.99Al | 8.9 | 1.3 |

## VI.  Conclusions

1.  There is a "break point" in the linear decrease of the lattice parameters, located between 125° and 100°K.

2.  Below the "break point" the "a" lattice parameter continues to decrease, but at a reduced rate.

3.  Below the "break point" the "c" lattice parameter remains nearly constant for the titanium-aluminum and the titanium-tin alloys and decreases at a reduced rate for the titanium-vanadium alloys.

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## Appendix A

### Raw Data (2θ Values)

The following tables contain the measured 2θ values
for each alloy and temperature. All listed values have
been corrected for the time-constant lag.

Table II

Raw Data, Ti-2.11V Alloy

2θ in Degrees

| hkl | 300°K | 250°K | 200°K | 160°K | 125°K |
|-----|-------|-------|-------|-------|-------|
| 110 | 62.97 | 63.01 | 63.04 | 63.07 | 63.08 |
| 103 | 70.57 | 70.59 | 70.60 | 70.61 | 70.61 |
| 112 | 76.21 | 76.24 | 76.25 | 76.27 | 76.29 |
| 201 | 77.42 | 77.45 | 77.48 | 77.50 | 77.51 |
| 203 | 102.29 | 102.35 | 102.42 | 102.46 | 102.47 |
| 211 | 109.17 | 109.26 | 109.32 | 109.37 | 109.39 |
| 114 | 114.13 | 114.19 | 114.24 | 114.28 | 114.32 |
| 213 | 139.49 | 139.60 | 139.70 | 139.79 | 139.86 |

| hkl | 100°K | 85°K | 40°K | 5°K |
|-----|-------|------|------|-----|
| 110 | 63.08 | 63.08 | 63.09 | 63.10 |
| 103 | 70.62 | 70.62 | 70.63 | 70.63 |
| 112 | 76.30 | 76.30 | 76.31 | 76.32 |
| 201 | 77.52 | 77.52 | 77.53 | 77.53 |
| 203 | 102.48 | 102.48 | 102.49 | 102.49 |
| 211 | 109.40 | 109.40 | 109.41 | 109.41 |
| 114 | 114.33 | 114.34 | 114.35 | 114.36 |
| 213 | 139.91 | 139.92 | 139.97 | 139.98 |

Table III

Raw Data, Ti-4.04V Alloy

$2\theta$ in Degrees

| hkl | 300°K | 250°K | 200°K | 160°K | 125°K |
|-----|-------|-------|-------|-------|-------|
| 110 | 63.02 | 63.04 | 63.06 | 63.08 | 63.09 |
| 103 | 70.55 | 70.57 | 70.58 | 70.58 | 70.59 |
| 112 | 76.25 | 76.27 | 76.28 | 76.29 | 76.30 |
| 201 | 77.46 | 77.48 | 77.49 | 77.50 | 77.51 |
| 203 | 102.33 | 102.39 | 102.45 | 102.49 | 102.49 |
| 211 | 109.21 | 109.29 | 109.35 | 109.40 | 109.41 |
| 114 | 114.18 | 114.25 | 114.31 | 114.36 | 114.38 |
| 213 | 139.57 | 139.66 | 139.74 | 139.82 | 139.88 |

| hkl | 100°K | 85°K | 40°K | 5°K |
|-----|-------|------|------|-----|
| 110 | 63.10 | 63.10 | 63.10 | 63.10 |
| 103 | 70.59 | 70.59 | 70.60 | 70.60 |
| 112 | 76.31 | 76.31 | 76.31 | 76.32 |
| 201 | 77.51 | 77.51 | 77.52 | 77.52 |
| 203 | 102.50 | 102.49 | 102.50 | 102.51 |
| 211 | 109.42 | 109.42 | 109.43 | 109.43 |
| 114 | 114.39 | 114.39 | 114.40 | 114.41 |
| 213 | 139.92 | 139.93 | 139.99 | 140.00 |

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## Table IV

### Raw Data, Ti-1.37Sn Alloy

#### 2θ in Degrees

| hkl | 300°K | 250°K | 200°K | 160°K | 125°K |
|-----|-------|-------|-------|-------|-------|
| 110 | 62.85 | 62.89 | 62.93 | 62.97 | 62.99 |
| 103 | 70.31 | 70.33 | 70.39 | 70.39 | 70.41 |
| 112 | 76.04 | 76.07 | 76.10 | 76.13 | 76.14 |
| 201 | 77.25 | 77.29 | 77.33 | 77.36 | 77.39 |
| 203 | 102.06 | 102.12 | 102.18 | 102.22 | 102.26 |
| 211 | 108.94 | 109.02 | 109.09 | 109.15 | 109.20 |
| 114 | 113.83 | 113.90 | 113.96 | 114.01 | 114.06 |
| 213 | 139.05 | 139.16 | 139.26 | 139.35 | 139.43 |

| hkl | 100°K | 85°K | 40°K | 5°K |
|-----|-------|------|------|-----|
| 110 | 63.00 | 63.01 | 63.00 | 62.99 |
| 103 | 70.42 | 70.41 | 70.42 | 70.41 |
| 112 | 76.15 | 76.16 | 76.16 | 76.17 |
| 201 | 77.41 | 77.42 | 77.42 | 77.41 |
| 203 | 102.29 | 102.30 | 102.29 | 102.30 |
| 211 | 109.21 | 109.21 | 109.22 | 109.23 |
| 114 | 114.09 | 114.10 | 114.09 | 114.10 |
| 213 | 139.48 | 139.48 | 139.49 | 139.50 |

## Table V

### Raw Data, Ti-2.56Sn Alloy

### 2θ in Degrees

| hkl | 300°K | 250°K | 200°K | 160°K | 125°K |
|-----|-------|-------|-------|-------|-------|
| 110 | 62.85 | 62.89 | 62.92 | 62.96 | 62.98 |
| 103 | 70.28 | 70.31 | 70.34 | 70.36 | 70.38 |
| 112 | 76.02 | 76.05 | 76.07 | 76.09 | 76.11 |
| 201 | 77.26 | 77.30 | 77.33 | 77.36 | 77.38 |
| 203 | 102.03 | 102.09 | 102.16 | 102.20 | 102.25 |
| 211 | 108.93 | 109.01 | 109.08 | 109.14 | 109.19 |
| 114 | 113.78 | 113.84 | 113.89 | 113.96 | 114.02 |
| 213 | 138.98 | 139.09 | 139.21 | 139.30 | 139.38 |

| hkl | 100°K | 85°K | 40°K | 5°K |
|-----|-------|------|------|-----|
| 110 | 62.99 | 62.99 | 62.99 | 62.99 |
| 103 | 70.38 | 70.38 | 70.38 | 70.38 |
| 112 | 76.12 | 76.12 | 76.13 | 76.13 |
| 201 | 77.40 | 77.41 | 77.41 | 77.42 |
| 203 | 102.22 | 102.23 | 102.23 | 102.23 |
| 211 | 109.20 | 109.20 | 109.21 | 109.22 |
| 114 | 114.03 | 114.04 | 114.04 | 114.05 |
| 213 | 139.43 | 139.44 | 139.45 | 139.46 |

## Table VI

### Raw Data, Ti-3.10Al Alloy

### 2θ in Degrees

| hkl | 300°K | 250°K | 200°K | 160°K | 125°K |
|-----|-------|-------|-------|-------|-------|
| 110 | 63.12 | 63.15 | 63.18 | 63.20 | 63.22 |
| 103 | 70.58 | 70.61 | 70.65 | 70.67 | 70.70 |
| 112 | 76.37 | 76.41 | 76.44 | 76.46 | 76.49 |
| 201 | 77.60 | 77.63 | 77.67 | 77.70 | 77.72 |
| 203 | 102.55 | 102.61 | 102.67 | 102.71 | 102.74 |
| 211 | 109.50 | 109.57 | 109.64 | 109.69 | 109.75 |
| 114 | 114.33 | 114.39 | 114.46 | 114.51 | 114.56 |

| hkl | 100°K | 85°K | 40°K | 5°K |
|-----|-------|------|------|-----|
| 110 | 63.23 | 63.24 | 63.24 | 63.25 |
| 103 | 70.71 | 70.71 | 70.70 | 70.71 |
| 112 | 76.50 | 76.51 | 76.52 | 76.51 |
| 201 | 77.74 | 77.74 | 77.75 | 77.76 |
| 203 | 102.77 | 102.78 | 102.78 | 102.78 |
| 211 | 109.78 | 109.79 | 109.80 | 109.81 |
| 114 | 114.59 | 114.59 | 114.60 | 114.60 |

## Table VII

### Raw Data, Ti-4.99Al Alloy

$2\theta$ in Degrees

| hkl | 300°K | 250°K | 200°K | 160°K | 125°K |
|-----|-------|-------|-------|-------|-------|
| 110 | 63.3q | 63.34 | 63.39 | 63.42 | 63.45 |
| 103 | 70.67 | 70.70 | 70.74 | 70.77 | 70.80 |
| 112 | 76.49 | 76.53 | 76.58 | 76.63 | 76.65 |
| 201 | 77.79 | 77.84 | 77.90 | 77.94 | 77.98 |
| 203 | 102.69 | 102.76 | 102.80 | 102.85 | 102.90 |
| 211 | 109.78 | 109.87 | 109.97 | 110.04 | 110,10 |
| 114 | 114.48 | 114.55 | 114.62 | 114.67 | 114.73 |

| hkl | 100°K | 85°K | 40°K | 5°K |
|-----|-------|------|------|-----|
| 110 | 63.47 | 63.47 | 63.48 | 63.48 |
| 103 | 70.81 | 70.81 | 70.80 | 70.80 |
| 112 | 76.57 | 76.67 | 76.68 | 76.68 |
| 201 | 77.99 | 77.99 | 78.00 | 78.01 |
| 203 | 102.91 | 102.91 | 102.92 | 102.93 |
| 211 | 110.15 | 110.15 | 110.16 | 110.17 |
| 114 | 114.75 | 114.76 | 114.76 | 114.77 |

## Appendix B

### Probable Error in "a" and "c" Values

#### Ti-2.11V

| Temp (°K) | "a" (Å) | "c" (Å) |
|---|---|---|
| 300 | 0.0004 | 0.0006 |
| 250 | 0.0003 | 0.0005 |
| 200 | 0.0002 | 0.0003 |
| 160 | 0.0002 | 0.0003 |
| 125 | 0.0002 | 0.0003 |
| 100 | 0.0002 | 0.0004 |
| 85 | 0.0003 | 0.0004 |
| 40 | 0.0004 | 0.0006 |
| 5 | 0.0004 | 0.0007 |

#### Ti-4.04V

| Temp (°K) | "a" (Å) | "c" (Å) |
|---|---|---|
| 300 | 0.0004 | 0.0007 |
| 250 | 0.0003 | 0.0005 |
| 200 | 0.0003 | 0.0005 |
| 160 | 0.0005 | 0.0007 |
| 125 | 0.0004 | 0.0006 |
| 100 | 0.0004 | 0.0006 |
| 85 | 0.0004 | 0.0007 |
| 40 | 0.0004 | 0.0007 |
| 5 | 0.0004 | 0.0007 |

#### Ti-1.37Sn

| Temp (°K) | "a" (Å) | "c" (Å) |
|---|---|---|
| 300 | 0.0004 | 0.0006 |
| 250 | 0.0004 | 0.0006 |
| 200 | 0.0004 | 0.0006 |
| 160 | 0.0004 | 0.0006 |
| 125 | 0.0004 | 0.0007 |
| 100 | 0.0005 | 0.0007 |
| 85 | 0.0006 | 0.0009 |
| 40 | 0.0004 | 0.0007 |
| 5 | 0.0005 | 0.0007 |

#### Ti-2.56Sn

| Temp (°K) | "a" (Å) | "c" (Å) |
|---|---|---|
| 300 | 0.0004 | 0.0007 |
| 250 | 0.0004 | 0.0006 |
| 200 | 0.0003 | 0.0005 |
| 160 | 0.0004 | 0.0007 |
| 125 | 0.0005 | 0.0008 |
| 100 | 0.0006 | 0.0009 |
| 85 | 0.0006 | 0.0010 |
| 40 | 0.0006 | 0.0009 |
| 5 | 0.0006 | 0.0010 |

#### Ti-3.10Al

| Temp (°K) | "a" (Å) | "c" (Å) |
|---|---|---|
| 300 | 0.0004 | 0.0007 |
| 250 | 0.0005 | 0.0007 |
| 200 | 0.0004 | 0.0006 |
| 160 | 0.0004 | 0.0006 |
| 125 | 0.0002 | 0.0004 |
| 100 | 0.0002 | 0.0004 |
| 85 | 0.0003 | 0.0005 |
| 40 | 0.0003 | 0.0006 |
| 5 | 0.0002 | 0.0004 |

#### Ti-4.99Al

| Temp (°K) | "a" (Å) | "c" (Å) |
|---|---|---|
| 300 | 0.0004 | 0.0006 |
| 250 | 0.0004 | 0.0006 |
| 200 | 0.0005 | 0.0008 |
| 160 | 0.0005 | 0.0008 |
| 125 | 0.0005 | 0.0008 |
| 100 | 0.0006 | 0.0009 |
| 85 | 0.0006 | 0.0010 |
| 40 | 0.0006 | 0.0010 |
| 5 | 0.0006 | 0.0010 |

Appendix C

## Alloy Analysis

Weight Percent

| Ti-2.11V | Ti-4.04V |
|----------|----------|
| 0.075 Fe | 0.035 Fe |
| 0.007 $N_2$ | 0.006 $N_2$ |
| 0.024 C | 0.026 C |
| 0.094 $O_2$ | 0.090 $O_2$ |

| Ti-1.37Sn | Ti-2.56Sn |
|-----------|-----------|
| 0.027 Fe | 0.030 Fe |
| 0.008 $N_2$ | 0.006 $N_2$ |
| 0.024 C | 0.024 C |
| 0.093 $O_2$ | 0.072 $O_2$ |

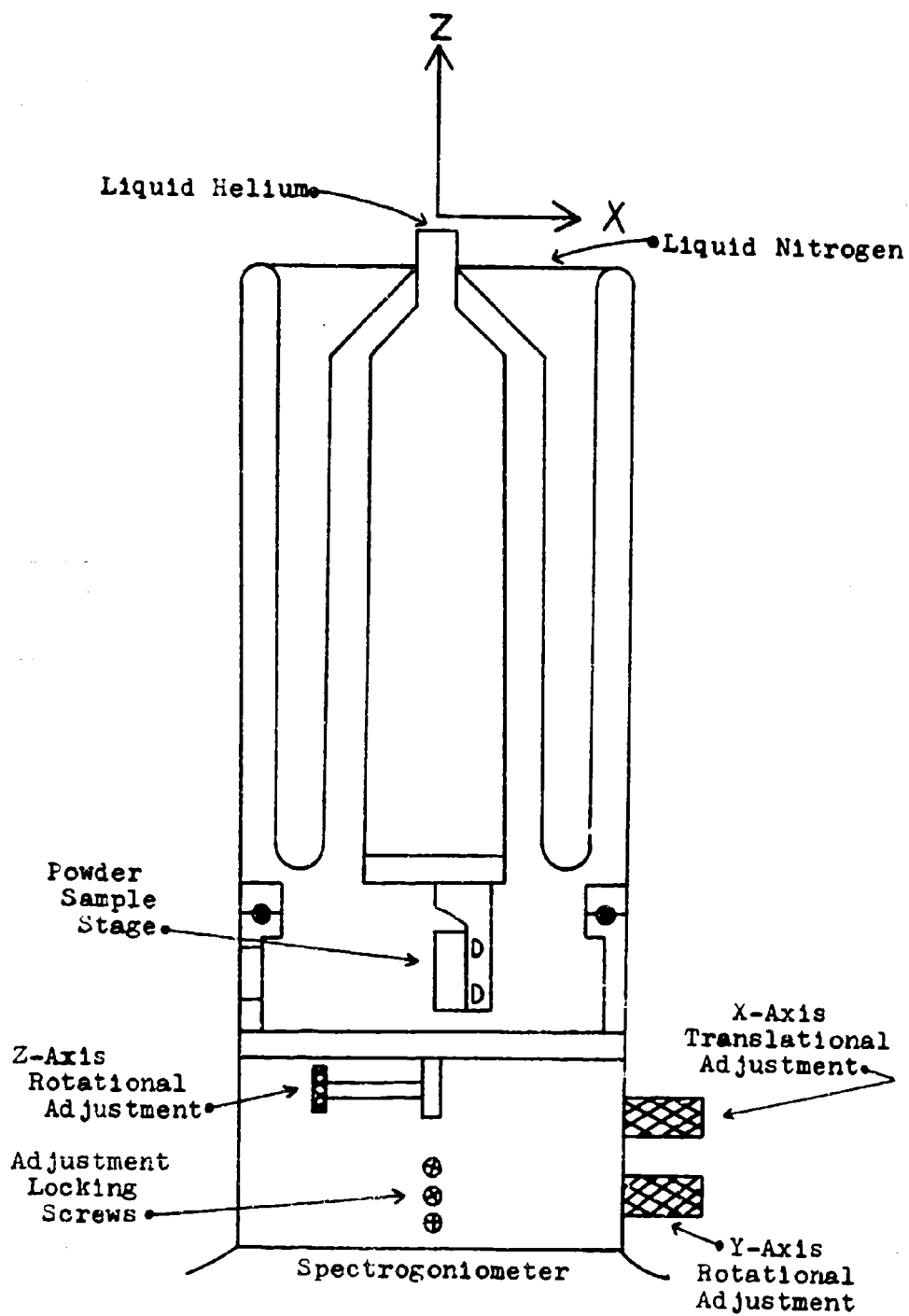| Ti-3.10Al | Ti-4.99Al |
|-----------|-----------|
| 0.039 Fe | 0.089 Fe |
| 0.007 $N_2$ | 0.005 $N_2$ |
| 0.026 C | 0.024 C |
| 0.076 $O_2$ | 0.104 $O_2$ |

Fig. 8.   MRC X-86GC Cryogenic Attachment

## Appendix E

## Lattice Parameters for Titanium
## Alloys at Room Temperature

Presented in the following tables are the room temperature lattice parameter values calculated in this study and by other investigators. Ebneter's values are from reference 3, and all other values are from reference 7.
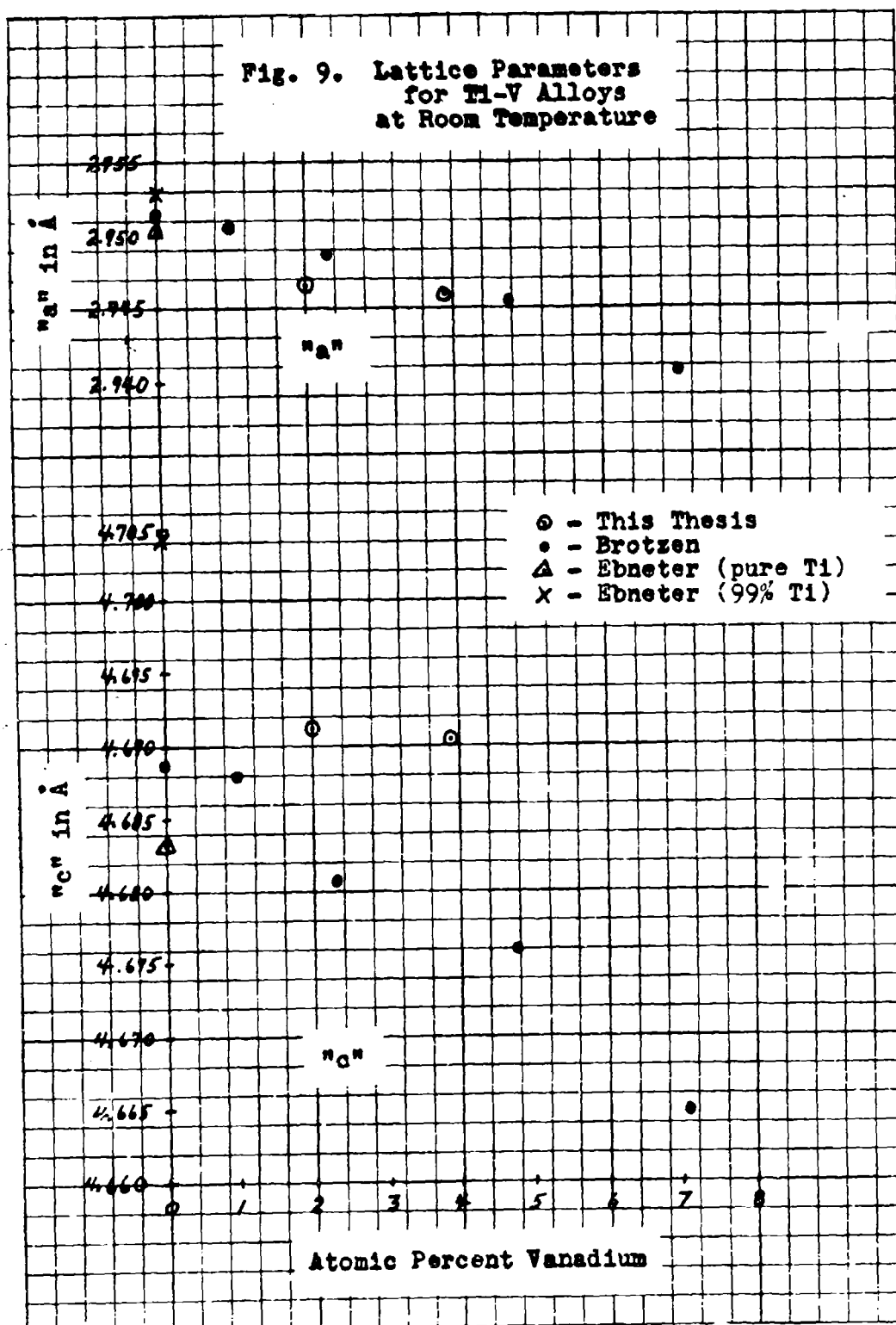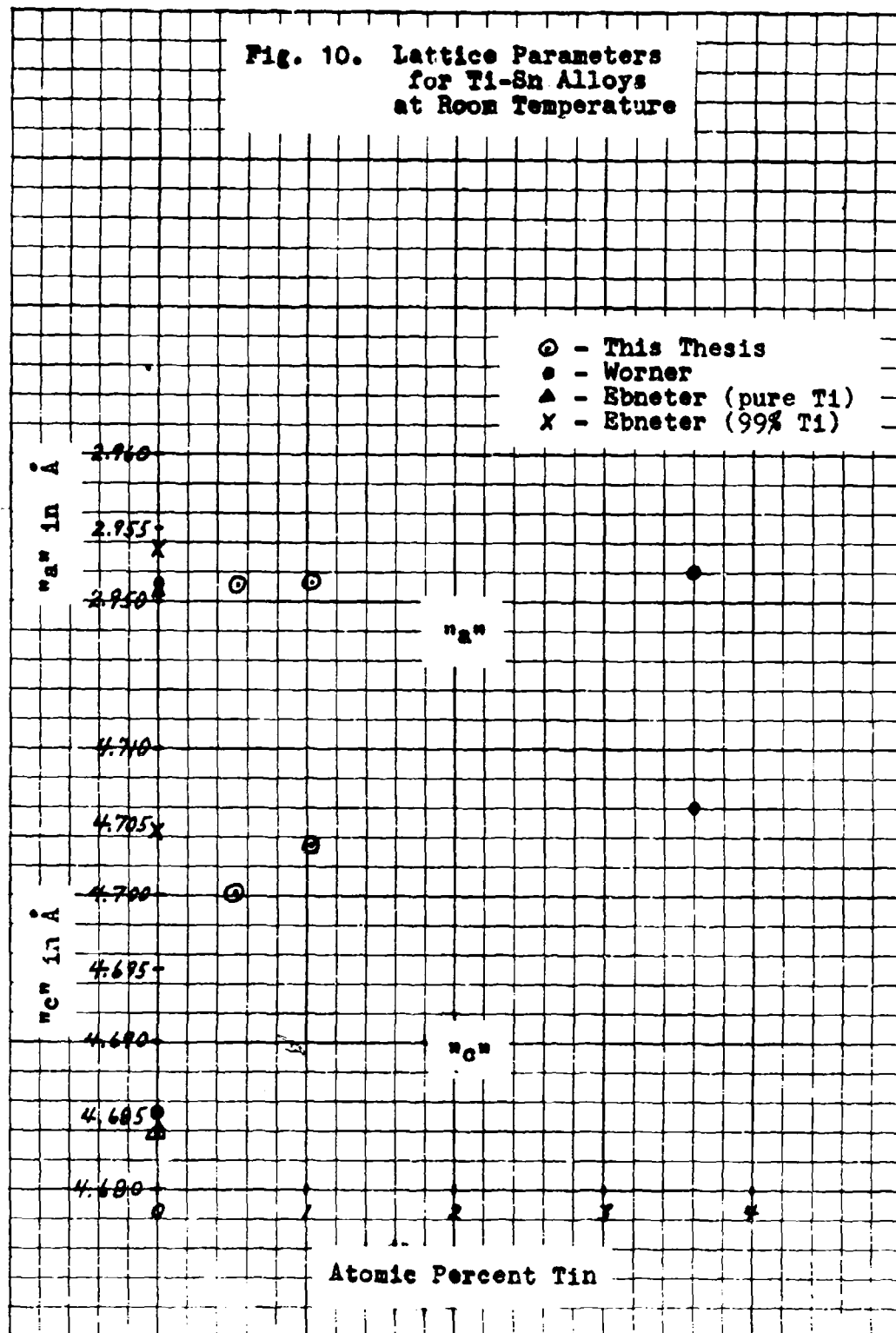
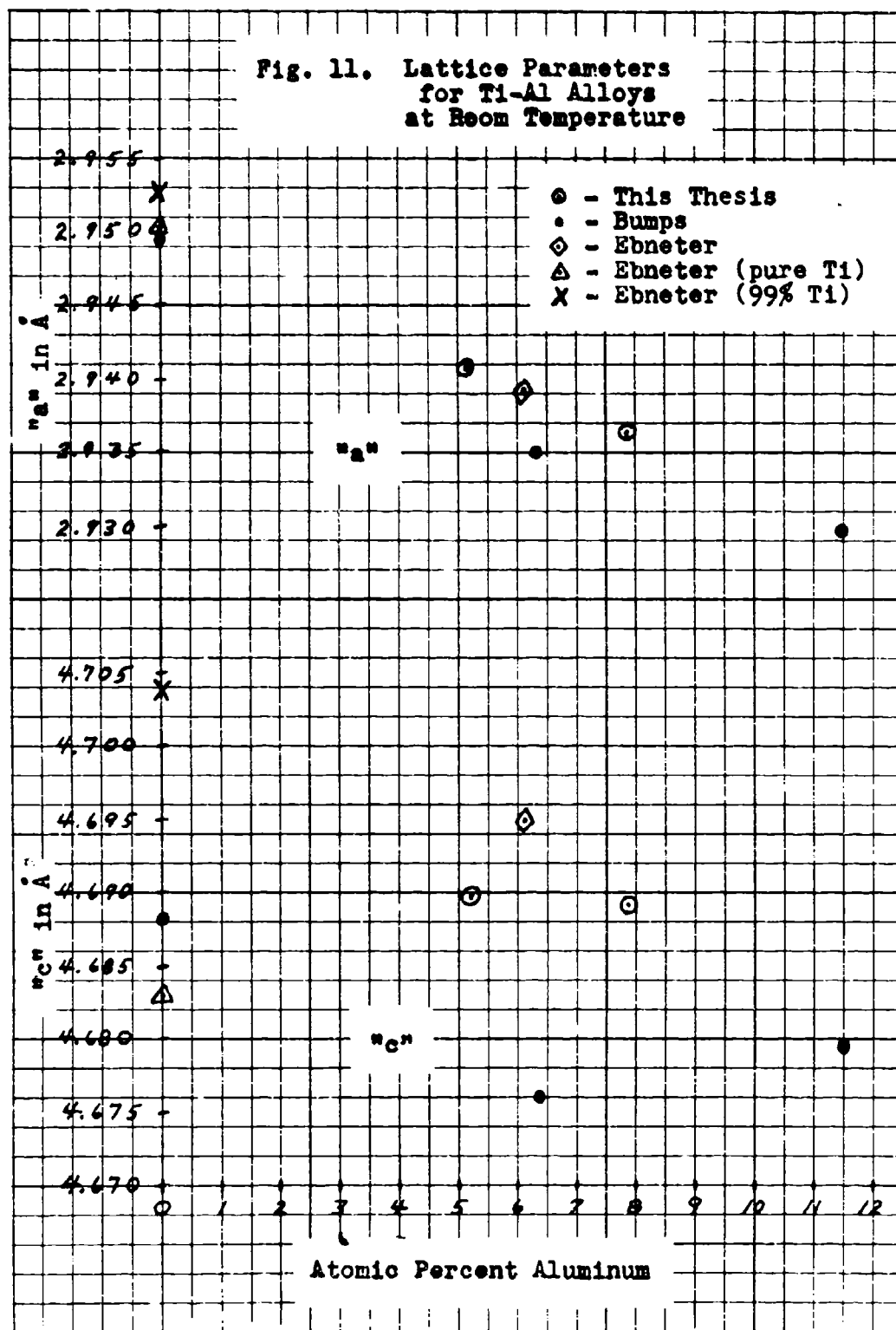Fig. 9. Lattice Parameters for Ti-V Alloys at Room Temperature

Fig. 10. Lattice Parameters
for Ti-Sn Alloys
at Room Temperature

⊙ - This Thesis
● - Worner
▲ - Ebneter (pure Ti)
X - Ebneter (99% Ti)

Atomic Percent Tin

Fig. 11. Lattice Parameters for Ti-Al Alloys at Room Temperature

## Vita

Roy George Inness, Jr. was born 1 October 1937 in Donaldsonville, Louisiana, the son of Roy George Inness and Rosemary Allen Inness. He graduated from Ascension High School in 1955 and received his Bachelor of Science degree from the United States Air Force Academy in 1959. After completing pilot training and F-100 advanced flying school, he was assigned to the 615$^{th}$ Tactical Fighter Squadron (F-100) at England AFB, Louisiana. In January 1963 he was assigned to the 416$^{th}$ Tactical Fighter Squadron at Misawa AB, Japan. He redeployed with the 416$^{th}$ Tactical Fighter Squadron to England AFB, Louisiana in June 1964 and to the Republic of Vietnam in November 1965. After completing 217 combat missions in Vietnam, he was assigned to AFIT in August 1966.

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**13. ABSTRACT**

The lattice parameters of six binary titanium alloys (3.10Al, 4.99Al, 2.11V, 4.04V, 1.37Sn, and 2.56Sn) were determined over temperature range 300° to 5°K using the powder x-ray diffraction technique. Both the "a" and "c" lattice parameters decreased linearly from 300°K to a "break point" between 125° and 100°K. The "a" lattice parameter then continued to decrease, at a reduced rate, from the "break point" to 5°K. Below the "break point" the "c" lattice parameter either remained constant or continued to decrease at a reduced rate, depending on the alloy.

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